Stable Materials and Bonding Techniques for Space-Based Optical Systems

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***Abstract*- Advances in material science have expanded the list of materials and bonding techniques available that can be used to design complex structures with exceptional quality and reliability for use in space-based missions such as LISA, TPF, SIM, BBO, or the GRACE follow-on mission. These missions require complex optical systems made from materials and bonding techniques that must meet unprecedented dimensional stability requirements. In some cases, the intrinsic dimensional stability needs to be better than 100 fm/$\sqrt{\text{Hz}}$ at Fourier frequencies as low as 0.1 mHz, or maintain a relative length stability less than one part per million over the lifetime of the mission. While there are materials that are thought to meet these requirements, few have been tested to these demanding levels. We present preliminary results on the dimensional stability of Zerodur, Silicon Carbide, and Super Invar at sub-pm levels, sufficient for most in-band applications. In addition, these missions rely on bonding techniques to assemble structures such as the telescope or optical bench. A new technique know as hydroxide bonding was originally developed for glass-glass bonds for the GPB mission, but has since been applied to other materials such as Zerodur, Silicon Carbide, and Super Invar. We will also report on first bonding strength measurements of hydroxide bonding between various materials and compare the dimensional stability of these bonds with the stability of optical contacts. This work is supported by NASA/OSS grant APRA04-0095-0007.**

I. Introduction

Many future space missions will use interferometry to make new discoveries about the universe. The LISA mission will use laser interferometry to measure gravitational waves produced from a range of sources including merging super-massive black holes and neutron star binaries. The entire Navigator program, from the Keck Interferometer to the TPF-I mission, uses different types of interferometers to increase their angular resolution [1]. Even the GRACE follow-on mission will use laser interferometry to map the Earth's gravitational field [2]. Each of these missions have demanding tolerances on the dimensional length changes for critical optical components. As an example, consider the telescope support structure for the LISA mission. The separation between the primary and secondary mirror is approximately 50 cm, but this distance cannot change by more than 1.2 μm over the lifetime of the mission while maintaining a noise spectrum below 1 pm/$\sqrt{\text{Hz}}$ at Fourier frequencies above 3 mHz. Advances in thermal insulation and computer models allow engineers to design an environment that will reduce thermally driven length changes, but our knowledge about the intrinsic length stabilities at the pico or femtometer level due to stress releases, creep, or aging of the material is limited.

In addition to being dimensionally stable, the bonding techniques used to adhere optical components must be able to withstand the harsh thermal and vibrational conditions endured during launch while maintaining their dimensional stability. Two such bonding techniques are optical contacting and hydroxide-catalysis bonding. Optical contacting occurs when two materials are polished smooth and flat enough and pressed together. The materials bond to each other through Van der Waals forces. If the surfaces have the same index of refraction, then the surfaces effectively disappear. The bond will break under thermal stresses, especially if the materials have significantly different coefficients of thermal expansion (CTE). The other technique, known as hydroxide bonding, has shown to offer versatility when adhering optical components of differing materials while providing superior bond strength when compared to optical contacting. This technique can be used to bond glass pieces to various types of materials, ranging from silicon carbide (SiC), to Super Invar, to other glass materials. While many properties of hydroxide bonding have been studied for glass-glass bonds, virtually nothing has been studied concerning glass-SiC bonds. In this paper, we present upper limits on the dimensional stability of SiC, Super Invar, and Zerodur, all of which are commonly used materials for space-based interferometric missions. In addition, we expand upon existing glass-glass bonds using the hydroxide bonding method, as well as present new results for glass-SiC bonds.

II. Material Stability Measurements

A. Experimental Setup

To test the dimensional stability of a material, it first needs to be thermally isolated from the outside. To do this, 5 cylindrical layers of gold-coated stainless steel are placed inside a vacuum chamber. Macor spacers are used to separate each

cylindrical layer from the next while providing support to the overall structure. This provides a temperature stability inside the thermal shields of less than a few tens of microKelvin at room temperature for frequencies above 1 mHz.

The material to be tested is used as a spacer for an optical cavity. Fused silica mirrors with anti-reflective coatings are then contacted to the spacer by use of either optical contacting or hydroxide bonding. Two cavities are then placed inside the thermal shields. The frequencies of two 1064 nm Nd:YAG lasers are stabilized to the cavities. To stabilize the laser to the resonance of each cavity, we use a modulation/demodulation technique commonly known as the Pound-Drever-Hall (PDH) technique [3]. In this technique, a laser beam is passed through a Faraday isolator to prevent instabilities in the laser that can occur from the back-reflections of optical components. The beam then passes through an electro-optic modulator (EOM) where the carrier phase is modulated with an RF frequency, usually between 10-30 MHz. The beam then passes through a polarizing beam splitter, a quarter-wave plate, and a series of lenses to match the spatial mode of the beam to the resonance mode of the cavity. Any light reflected from the cavity will be received on a photodiode, where it is turned into amplitude modulation that creates an oscillating photo-current. This signal is then demodulated with the phase modulation frequency produced from the EOM. The demodulated signal will be proportional to the difference between the laser frequency and the resonance frequency of the optical cavity as long as the laser frequency is within a linewidth of one of the cavity resonances. The demodulated signal is then amplified and fed back to the frequency actuators of the lasers. The lasers have a slow and fast frequency actuator. Using both these actuators, we are able to keep the frequency of the laser locked to the resonance frequency of the optical cavity. Any length changes in the cavity will produce a change in the frequency of the laser. By comparing the laser frequencies, we are able to determine the relative changes in length of the cavities.

In addition to the first vacuum tank, we have another chamber which is used to test multiple materials at the same time. Within this chamber are thermal shields made from an aluminized PET material. This provides a cost-effective alternative to gold-coated stainless steel. Due to space limitations, a fiber optic cable is used to get the laser beam from the table to the tank.

B. Optical Cavities

1) Zerodur: To find our noise floor, we first made two cavities by optically contacting mirrors to a Zerodur spacer. The material and bonding technique used to bond the mirrors to the substrate were chosen because of their low noise characteristics. The results are presented in Fig. 1 [4]. The lower dashed curve corresponds to the noise limit the pre-stabilization cavities must meet for the LISA mission, while the dashed upper curve corresponds to the noise limit the telescope support structure must meet. These curves are provided as a reference for the different material stabilities. We then use one of these cavities as our reference for other measurements and replace the other

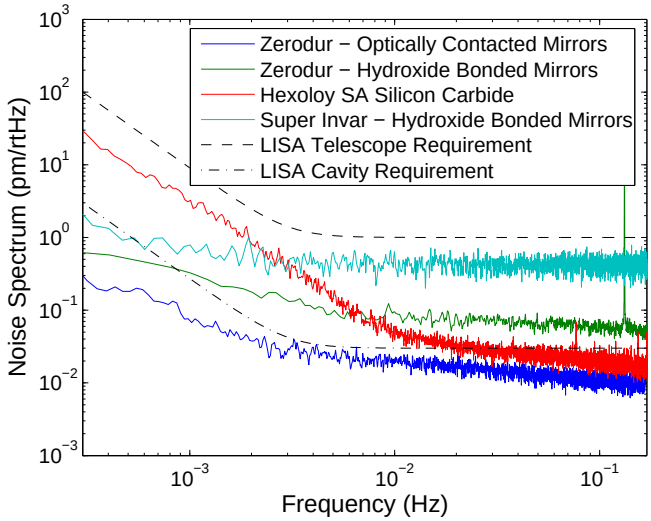

Fig. 1. The relative dimensional stability of Zerodur with optically contacted and hydroxide bonded mirrors, SiC with optically contacted mirrors, and Super Invar with hydroxide bonded mirrors. The LISA telescope and cavity requirement curves are provided as a reference.

cavity with a different material or bonding technique to be studied.

For a bond to form using the hydroxide bonding technique, the OH^{-} ions present in the solution act to etch the surfaces of the materials, resulting in a release of silicate ions which decreases the pH value [5]. Once the pH falls below a value of approximately 11, the silicate ions dissociate and reform into siloxane chains. As the water dehydrates, the siloxane chains bond together to form the resulting bond at the interface. While the hydroxide bonding method may provide significantly stronger bonds when compared to optical contacting, the bond itself may add noise to the dimensional stability of the cavity. This may be due to the fact that optical contacting is a direct contact between the materials at the interface, while hydroxide bonding produces a very thin bond between the materials. To test this, we have constructed another cavity out of Zerodur using hydroxide bonding to adhere both mirrors. We can then compare these results with those initially obtained from the Zerodur spacers with optically contacted mirrors. Current results are shown in Fig. 1. It should be noted that these curves are upper limits on the dimensional stability and should decrease as noise sources within our system are improved.

2) Silicon Carbide: SiC has emerged as a promising material to be used in space-based missions. Its high thermal conductivity, exceptional strength, ease to machine, and light weight make it ideal for many applications. The thermal and mechanical properties of SiC provide exceptional thermal shock resistance and could reduce temperature gradients. While its CTE is roughly two orders of magnitude larger than Zerodur at room temperature, at cryogenic temperatures they are comparable, making it an ideal material for mirrors or optical benches to be used in infrared astronomy.

To make the SiC cavity, mirrors were optically contacted to a

spacer made of Hexoloy SA SiC whose ends were polished to a λ/8 global flatness with a 60-40 scratch dig. Preliminary results showed the cavity to drift at approximately 1.0 kHz/s, but began to slow down over the course of a few months to about 200-400 Hz/s. This change in drift is assumed to be from the annealing of the optical contacts and stresses released from the material [6]. At higher frequencies, the dimensional stability of SiC is comparable to that of Zerodur, but at lower frequencies, it is closer to one or two orders of magnitude greater (Fig. 1). This may be a thermal effect induced from the heating of the mirrors by the laser beam.

3) SuperInvar: Metals are often used for supporting structures. They are easy to machine, flexible, strong, and can be designed to have a low CTE. One such metal is Super Invar. Super Invar is an iron-nickel-cobalt alloy with a CTE of only $6 * 10^{-7}/K$ at room temperature and is often used in applications in which the dimensional stability is critical, but glasses such as ULE or Zerodur cannot be used. Unfortunately, the properties of Super Invar make it hard to polish sufficiently enough to obtain a significant bond using optical contacting. Other bonding techniques such as frit or anodic bonding, both of which require heating of the materials, may have produced better results, but the difference in CTEs of Super Invar and most glasses make it difficult to produce substantial bonding strengths if the substrate are too thick. For these reasons, hydroxide bonding was chosen to bond the mirrors to the Super Invar cavity.

After the cavity was made, it was placed in the vacuum tank with the PET thermal shields. As noted earlier, the primary difference between the setup with the PET thermal shields and that with the gold-coated thermal shields is that a fiber optic cable is used to get the laser beam from the optical bench to the vacuum chamber for the PET thermal shields. An upper bound for the dimensional stability is shown in Fig. 1. The current noise ceiling is most likely due to noise introduced from the fiber and should decrease as the system is improved.

III. Hydroxide Bonding Strength

Hydroxide bonding has shown its versatility by being capable of bonding glass to materials such as SiC, Super Invar, or even silver-plated PZTs. The hydroxide bonding process requires a choice of hydroxide, the hydroxide concentration, and how much of the solution to use. These parameters allow a tailor made bond to form for different materials. Each of these parameters can be varied to produce a bond of varying strength [7]. We have begun the process of studying these parameters to obtain an optimal bond strength. This versatility makes it a front-runner for many future applications in space-based interferometric missions. Although the noise spectrum of the bonds play a critical role in what materials and bonding techniques to use, if the bond cannot withstand the strenuous thermal and vibrational conditions endured during launch, then it cannot be used. For space-based interferometric missions, a typical optical component has an approximate bond area of 100 mm^2, mass of 10 mg, and is subject to $35 * g$ accelerations during launch. This results in a requirement that the bonds be able to withstand on order of tens of kPa.

For our experiments, BK7 glass and Hexoloy SA SiC were used as bonding materials. Both glass-glass bonds and glass-SiC bonds were made using the hydroxide bonding technique. The BK7 glass pieces were 12.7 mm in diameter and 10 mm thick. One side was polished to a λ/8 flatness with a 20-10 scratch/dig on one side, and a ground polish on the other side. This allowed us to test bonds with both rough and smooth faces. The SiC pieces were 50.8 mm on a side and 6.35 mm thick with a λ/8 flatness and 60-40 scratch-dig on both sides. This allowed us to test multiple bonds on one SiC piece.

To test the shear strength of the bonds, we constructed a lever arm that could hold both the glass-glass bonded pieces as well as the glass-SiC bonded pieces. The lever arm had a 5:1 torque advantage with a 1/8" stainless steel wire that was wrapped around the glass piece approximately 3 mm from the bond. A force was applied in a downward manner on the other side of the lever arm where a spring-based force meter that had a maximum force indicator was used to measure the applied force to the glass piece.

A. Glass to Glass Bonds

The dehydration of the water plays an important role in the hydroxide bonding process. Not only does it provide the change in pH necessary for the bonding to take place, but it allows the siloxane chains formed at the surface to entangle and form the rigid 3D structure that joins the two surfaces. It also plays another crucial role. If it takes a substantial amount of time for the bond to reach maximum strength due to the dehydration of the solution, then this can have an impact on the design and fabrication process. To determine the effects of dehydration time on the shear strength, multiple samples were prepared using 0.40 μL/cm^2 of a volumetric 1:4 sodium silicate (Sigma-Aldrich) to de-ionized water solution between two glass pieces using the unpolished sides, all with 1.27 cm^2 of bonding area. The bonded pieces stayed 24 hours in the clean room where they were prepared. One piece was then broken after this 24 hour period and the rest of the samples were put in an oven at 330 K to expedite the dehydration and were kept there until broken. Two bonded piece were kept in the clean room to determine the breaking strength effects of the elevated temperature within the oven. The results are shown in Fig. 2.

From Fig. 2 it is evident the dehydration time has little or no effect on the breaking strength and the bond reaches maximum strength after approximately 24 hours. After breaking the pieces that were left in the clean room for 11 days, they were found to have a similar breaking strength to the pieces left in the oven for the same amount of time. It should be noted that when breaking the bonded glass pieces, the failure was almost always a few millimeters above the bond where the wire on the apparatus applied the force on the glass, and not at the glass-glass interface.

It was shown in [7] that when the hydroxide concentration was varied, the breaking strength varied as well. Another

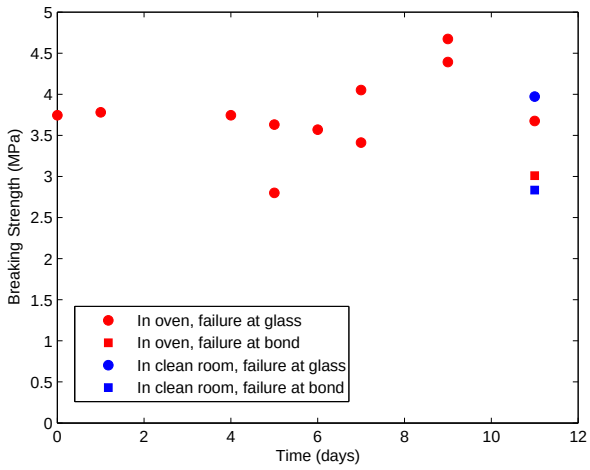

Fig. 2. Breaking strengths of glass-glass bonds after being kept in an oven at 330 K. The zero on the x-axis represents the time after the samples were in the clean room for 24 hours.

Concentration	μL Used	Breaking Strength(MPa)
1:4	0.25	2.34 (+0.24, -0.24)
1:4	0.50	3.19 (+0.35, -0.52)
1:4	0.75	4.65 (+2.98, -2.99)
1:4	1.00	4.69 (+0.40, -0.39)

TABLE I

BREAKING STRENGTHS AS A FUNCTION OF THE AMOUNT OF SODIUM SILICATE SOLUTION USED WITH ALL BONDING AREAS KEPT CONSTANT. THE AVERAGE IS GIVEN AS THE FIRST NUMBER IN THE THIRD COLUMN, WHILE THE NUMBERS IN THE PARENTHESIS PROVIDE THE RANGE.

parameter that may affect the shear strength is the amount of solution used per square centimeter. To test this, we used the same solution as described above and varied the amount used between the polished sides of the glass pieces. All bonded pieces were allowed to dry for at least 24 hours at room temperature. In this manner, the bonding area was kept constant and the amount of solution changed. The results are shown in Table 1.

From our results, there appears to be an increasing trend in the breaking strength of the glass as the amount of solution used increases. This may be due to the fact that more siloxane chains are created which, in turn, provides a stronger bond. In almost all tests, the point of failure was at the glass.

B. Glass to SiC Bonds

While many properties of glass-glass bonding have been studied using hydroxide bonding, as well as other methods, virtually nothing has been done concerning glass-SiC bonds. While it was possible to optically contact a glass piece to the SiC, the strength was negligible and could be broken off using your hands. Using hydroxide bonding, we were able to bond the BK7 glass to the SiC with appreciable strength. As in the previous section, we kept the bond area constant while varying the amount of solution used. In addition, we tested the bonded pieces using a dilution factor of both 1:4 and 1:6 of the sodium

Concentration	μL Used	Breaking Strength(MPa)
1:4	0.12	3.51 (+0.63, -0.40)
1:4	0.25	3.26 (+0.91, -0.86)
1:4	0.35	2.41 (+0.65, -0.66)
1:4	0.50	2.12 (+1.38, -1.11)
1:4	0.75	1.80 (+0.40, -0.28)
1:6	0.12	2.05 (+0.32, -0.33)
1:6	0.25	3.08 (+0.69, -1.15)
1:6	0.50	2.04 (+0.27, -0.54)
1:6	0.75	2.67 (+0.88, -1.66)

TABLE II

BREAKING STRENGTHS FOR GLASS-SIC BONDS BY VARYING THE AMOUNT OF SOLUTION USED, AS WELL AS VARYING THE DILUTION FACTOR.

Concentration	μL Used	Breaking Strength(MPa)
1:4	0.25	2.22 (+0.25, -0.42)
1:4	0.50	1.90 (+0.76, -0.76)
1:4	0.75	2.89 (+0.12, -0.12)

TABLE III

BREAKING STRENGTHS USING A ROUGH GLASS SURFACE WITH A DILUTION FACTOR OF 1:4.

silicate by volume. This was done because it is possible that by using a higher dilution factor fewer siloxane chains will be able to form and interlace at both surfaces. Our results are shown in Table 2.

Our results seem to show that for a 1:4 dilution factor, increasing the amount of solution used per square centimeter reduces the breaking strength, while there appears to be no appreciable trend in breaking strength using the 1:6 dilution factor. Roughly one third of the pieces tested using the 1:4 dilution factor broke at the glass, while the rest broke at the bond. Almost half of the pieces broke at the glass for the 1:6 dilution factor.

Another factor that may affect the breaking strength of the glass-SiC bonds is the surface profile. If the bonding surfaces are too rough, then the siloxane chains may not be able to bond to as much area since they may not be able to fill in the gaps, effectively leaving fewer places where the bond joins the two surfaces. To test this, we used the rough side of the glass pieces instead of the polished side as previously done. Our results are shown in Table 3.

While there doesn't seem to be any appreciable trend when the amount of solution is varied, the values are comparable to those when the polished side is used for both 1:4 and 1:6 dilution factors. It is important to note that while there is some variability in the measurements, even the lowest bonding strength for all measurements (both glass-glass and glass-SiC) was an order of magnitude or more than is required to withstand launch conditions.

IV. CONCLUSION

As space-based interferometric missions become increasingly sensitive, the choice of materials and bonding techniques used for critical optical components will become increasingly important. Both the dimensional stability of optical components and the strength of the bonds used to adhere them will need

to be chosen to meet the stringent demands of upcoming missions. Zerodur, SiC, and Super Invar are all materials that are commonly used. We have placed upper limits on their dimensional stabilities using various bonding techniques, as well as shown that hydroxide bonding can be used to provide a significantly stronger bond that optical contacting while adding little, if any, extra noise to the dimensional stability.

REFERENCES

[1] The search for terrestrial planets is the goal of NASA's Navigator program. This program include a series of ground-based and space-based interferometric telescopes. A good source for information about this quest is the Navigator website.

[2] Grace follow-on website

[3] E. Black, "An introduction to Pound-Drever-Hall laser frequency stabilization," *Am. Journ. Phys.*, vol. 69, no. 1, pp. 79-87, January 2001.

[4] R. J. Cruz and et al, "The LISA benchtop simulator at the University of Florida," *Class. Quantum Grav.*, vol. 23, pp. S761-S767, 2006.

[5] Gwo D-H, 2001, Ultra-precision and reliable bonding method, United States Patent no US 6 284 085 B1.

[6] J. W. Bertold III, S.F. Jacobs, M.A. Norton, "Dimensional Stability of Fused Silica, Invar, and Several Ultra-Low Thermal Expansion Materials," *Metrologia* 13, 9-16 (1977).

[7] E. J. Elliffe and et al "Hydroxide-catalysis bonding for stable optical systems for space," *Class. Quantum Grav.* 22, S257-S267, 2005.